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U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371

MPD309

U.S. APPLICATION NO. (If known, see 37 CFR 1.5

10/088980

INTERNATIONAL APPLICATION NO.
PCT/EP00/09677INTERNATIONAL FILING DATE
10/04/00PRIORITY DATE CLAIMED
10/7/99

TITLE OF INVENTION

STRUCTURED SURFACTANT SYSTEMS

APPLICANT(S) FOR DO/EO/US

HATCHMAN, Kevan; CROMBIE, Richard Llewelyn; HAWKINS, John

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ has been communicated by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
 - a. ☐ is attached hereto.
 - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ have been communicated by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

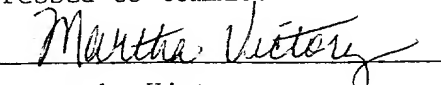
Items 11 to 20 below concern document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
14. ☐ A SECOND or SUBSEQUENT preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☐ Other items or information:

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page 1 of 2

I hereby certify that the application/correspondence attached hereto is being deposited with the U.S. Postal Service "Express Mail Post Office to Addressee" Service under 37 C.F.R. 1.10 on the date indicated below and is addressed to Commissioner for Patents, Washington, D.C. 20231


Martha Victory

26 March, 2002
Date of Deposit

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Hatchman et al.

Docket # *MPD 309*

Serial No. unk. (entry into National Stage in US
of PCT/EP00/09677

Filed: concurrently herewith

For: "STRUCTURED SURFACTANT
SYSTEMS"

March 7, 2002

Assistant Commissioner for Patents
Washington, D.C. 20231

Preliminary Amendment

Sir:

Prior to calculation of the filing fee in the above-captioned matter, please enter
the following amendments to the claims:

6) (AMENDED) A method of making a composition as claimed in claim 3 which
comprises forming an emulsion of ethylene glycol distearate, optionally in admixture
with a minor proportion of ethylene glycol monostearate, at a temperature above its
melting point, in a solution of from 18 to 28% by weight of a sodium C₁₀₋₁₈ alkyl 1 to
10 mole ethoxy sulphate and from 2 to 5 % by weight of sodium chloride and cooling
said emulsion.

0309

Christopher J. Whewell

Christopher J. Whewell, Reg. No. 37,469
c/o Huntsman Corp.
7114 North Lamar Boulevard
Austin, Tex. 78752
(512) 483-0919

Version with markings to show changes made

6) (AMENDED) A method of making a ~~pearliser suspension~~ composition as claimed in
~~any of claims 3 to 5~~ claim 3 which comprises forming an emulsion of ethylene glycol
distearate, optionally in admixture with a minor proportion of ethylene glycol
monostearate, at a temperature above its melting point, in a solution of from 18 to 28%
by weight of a sodium C₁₀₋₁₈ alkyl 1 to 10 mole ethoxy sulphate and from 2 to 5 % by
weight of sodium chloride and cooling said emulsion.

1003390-03469

STRUCTURED SURFACTANT SYSTEMS

The invention relates to a novel type of structured surfactant system. The novel system is especially suitable for suspending pearlising concentrates for incorporation into liquid formulations such as shampoos and toiletries to impart a nacreous iridescence which is attractive to consumers, and can mask inhomogeneities in the formulations.

Structured surfactant systems have the ability to suspend solid particles indefinitely in a pourable liquid. While the system is at rest it behaves like a solid gel holding the particles immobile, but the shear forces associated with pouring break the structure causing the suspension to flow like a mobile liquid. Known structured surfactant systems are either opaque interspersions of a surfactant mesophase with an aqueous phase or cloudy opalescent lamellar phases.

Pearlisers typically comprise small, thin, transparent platelet crystals which can be suspended in a parallel configuration. When so suspended light falling on the crystals undergoes complex multiple reflections within the substrate similar to those which occur in a pearl and giving rise to similar optical interference effects.

Natural pearls comprise alternate layers of calcium carbonate and protein. Artificial pearlisers include guanine/hypoxanthine crystals extracted from fish scales, mica, various salts of lead, zinc, mercury and bismuth (e.g. bismuth oxychloride), titanium oxide and various fatty acid derivatives such as magnesium stearate, coconut monoethanolamide, ethylene glycol distearate and ethylene glycol monostearate. Fish scale extracts are too expensive and the inorganic pearlisers are either too toxic for general use in toiletries e.g. lead, mercury, or relatively ineffective e.g. bismuth. The fatty acid derivatives are therefore now the most widely used pearlisers. In addition to the chemical nature and physical form of the pearliser the manner in which it is suspended has an important effect on its visual impact. Difficulty is sometimes encountered obtaining the desired effect when incorporating pearlisers into aqueous formulations.

Conventional fatty acid derived pearlisers are supplied as solids which are usually added to a heated formulation above their melting point and recrystallised in situ. The conditions of crystallisation and especially the amount and nature of the agitation applied must be carefully controlled in order to obtain an acceptable result. This

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makes it difficult to obtain consistent effects and renders solid pearlisers inconvenient to use.

Attempts have been made to prepare liquid concentrates or suspensions which can be added directly to shampoo formulations without heating. While more convenient for the user, such concentrates face the manufacturer with problems of obtaining a high and consistent pearl effect, similar to those which confront the user of conventional solid pearlisers. Difficulty is also encountered in maintaining the particles in stable suspension and preventing sedimentation.

We have now discovered that certain surfactants having a high solubility parameter such as alkyl ether sulphates at concentrations just below those which normally give rise to liquid crystal phases, e.g. typically 18 to 30% by weight, will form mobile, clear protolamellar L₁ phases in the presence of sufficiently high concentrations of electrolyte. These mobile phases are clear and optically isotropic and are typically less viscous than normal G-phases but have the capacity to form stable suspensions of solids such as pearlisers.

The protolamellar phase is apparently unique in being clear and optically isotropic while exhibiting the suspending properties of a structured surfactant system. We believe that protolamellar phases comprise oblate (disc) micelles which can adopt a parallel lamellar-like orientation. They may typically be identified by placing them between crossed polarisers and applying stress, e.g. by tapping or shaking, when flashes are observed as the system transiently forms an anisotropic structure.

When pearlisers are suspended in a protolamellar phase the structure promotes the parallel orientation of the pearlisng crystals which maximises the pearlescent effect, even in the absence of agitation. The concentrates are readily pourable but do not generally tend to separate on standing, and can easily be added to shampoo formulations, without heating, to give high and consistent pearlisng effects.

The compositions are particularly useful because the preferred surfactant, alkyl ether sulphate, is the most commonly used ingredient of shampoo formulations on account of it's skin mildness and comparatively good foaming properties. Thus the concentrate can be included in shampoo formulations without introducing anything extraneous to the formulator's requirements. The optical clarity of the protolamellar phase permits the pearlescent effect to be clearly observed without being obscured by surfactant structures as occurs when any hitherto known structured surfactant is used.

When alkyl ether sulphates or similar water soluble surfactants are dissolved in water at relatively low concentrations they form a clear micellar solution (L1 phase) with the surfactant molecules arranged in spherical clusters (micelles). As the concentration is increased the micelles form rods (prolate micelles) of increasing length, and the viscosity increases. Further increases of concentration are accompanied by an anomalous fall in viscosity as the rods become aligned. The aligned rod micellar or protohexagonal system is optically clear, pourable and may exhibit a hexagonal symmetry when examined by small angle x-ray diffraction (SAX), giving a first order peak of momentum transfer vector Q_1 , and, if the system is sufficiently well defined, one or more, progressively smaller, higher order peaks at Q values in the ratios:

$$Q_2 = Q_1 \sqrt{3} ; Q_3 = 2Q_1$$

These ratios are diagnostic of hexagonal symmetry.

When the concentration is further increased the length of rods increases indefinitely and the immobile M phase is formed. This is typically observed at concentrations of about 30% by weight surfactant. The M phase also shows SAX peaks in the ratios characteristic of hexagonal symmetry. It typically resembles a curdy or gelatinous solid or very viscous mucous like fluid. The normal lamellar, or G phase is typically formed when the concentration of the alkyl ether sulphate is increased to about 55 to 60% by weight. It is a mobile, birefringent liquid crystal with sheer dependent viscosity and lamellar symmetry, revealed by SAX peaks with the characteristic ratios $Q_2 = 2Q_1$, ; $Q_3 = 3Q_1$. The peaks indicate structure with a repeat spacing ($d = \frac{2\pi}{Q_1}$) of about 3 to 5 nm. Although the normal G-phase has a yield point and is relatively mobile compared with the M-phase it is not generally suitable for use as a suspending medium for solids, since the presence of appreciable amounts of suspended solid renders it unpourable. The high surfactant concentration is a further disadvantage.

However, when sufficient electrolyte is added to a micellar or protohexagonal solution of alkyl ether sulphate there appears to be a change from prolate to oblate micelles and corresponding shift from hexagonal to lamellar symmetry with a d spacing typically in the range 6 to 15 nm. Such phases are able to suspend solids and remain readily pourable.

Our invention further provides a suspension of solid particles in an aqueous surfactant, wherein said surfactant is in a protolamellar phase. In a preferred embodiment the suspended particles comprise particles of a pearliser.

The surfactant may comprise minor amounts of other surfactant, especially anionic, non-ionic or amphoteric surfactants, for example alkyl sulphates, alkyl benzene sulphonates, paraffin sulphonates, olefin sulphonates, alkyl sulpho-succinates, soaps, taurides, isethionates, alkyl ethoxylates, fatty acid ethoxylates, alkyl glyceryl ethoxylates, alkyl carbohydrate ethoxylates, anine oxides or betaines. However it is generally preferred to use essentially only alkyl ether sulphate.

The electrolyte is preferably sodium chloride, but could be, for example, sodium carbonate, sodium citrate, sodium tripolyphosphate, sodium hydroxide or any other salt or base which tends to salt alkyl ether sulphates out of solution. The cation of the electrolyte may be sodium, potassium lithium, ammonium or, less preferably, an amine such as alkanolamine or a mixture of any of the foregoing.

The choice of electrolyte and of auxiliary surfactant, if any, may be conditioned by what ingredients the formulator, to whom the concentrate is to be supplied, wishes to include in the end formulations, or can tolerate.

Generally the surfactant is used in a total concentration which, in the absence of electrolyte, would correspond to a clear L₁ phase or protohexagonal phase. Typically the electrolyte-free surfactant/water system would be unstructured or would exhibit hexagonal symmetry under SAX. The concentrations required vary with different surfactants but are generally in the range 17 to 30% based on the total weight of the composition, more usually 19 to 28%.

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The amount of electrolyte is sufficient to convert the ether sulphate/water mixture from an optically isotropic, clear, L_1 phase or a protohexagonal phase exhibiting hexagonal symmetry to a clear, protolamellar phase, typically exhibiting lamellar symmetry, under SAX. The amount required will depend on the nature and concentration of the surfactant. Typically it will be between 1 and 10% based on the total weight of the composition, more usually 2 to 5%. Sodium citrate is more expensive than sodium chloride and is most effective at concentrations of 4 to 10% but permits higher loading of pearlisers.

The pearlisers may be any of those previously discussed herein including natural pearl and inorganic pearlisers but is preferably a fatty acid derivative especially a mixture of ethylene glycol distearate and ethylene glycol monostearate.

The pearlisers may be dispersed in the aqueous structure surfactant system e.g. by gently stirring, but in the case of the fatty acid derivatives are preferably prepared in situ by heating above their melting point, e.g. temperatures between 65 and 80°C, dispersing the liquid pearlisers in the structured surfactant system, preferably with sufficient stirring to form droplets of from 1 to 60 microns, e.g. 15 to 50 microns, and cooling to ambient temperature. Preferably cooling is relatively slow e.g. the mixture is allowed to cool naturally. The amount of pearlisers can be varied considerably, the main constraint on the upper limit being the viscosity.

The amount of pearlisers should not be so high as to render the product unpourable, or unacceptably viscous. We prefer on economic grounds that the pearlisers are present in amounts greater than suspending surfactant. Generally pearlisers may be present in amounts ranging from 5% up to about 60% e.g. 10 to 45% more preferably 15 to 30%, especially 18 to 28% of the total weight of the mixture. The ratio of pearlisers to surfactant is preferably from 0.6 to 2 e.g. 1.2 to 1.3 by weight.

Ancillary ingredients suitable for inclusion in toiletries could in principle be included, but are usually omitted to avoid unduly constraining the customer with regard to its freedom to formulate. However, it is generally desirable to include small amounts of preservative, such as formaldehyde or other microbiocidally and/or fungicidally active material.

The novel structured, protolamellar phase may be used to suspend solids other than pearlisers, e.g. builders such as zeolite or phosphate for use in detergent. They may also suspend bentonite or calcite. They may suspend rock cuttings and/or weighting

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agents for use a drilling muds. They can suspend agricultural pesticides or dyes or pigments, dicalcium phosphate for use in toothpaste or ammonium polyphosphate for fire retardants.

The invention is illustrated by the following examples:-

The method comprised of the following steps:

- i) Charge water and heat to 75-80°C.
- ii) Add NaCl and dissolve with mixing.
- iii) Add 80-90 % of SLES charge and mix until homogeneous.
- iv) Add EGDS and mix until a homogeneous emulsion is formed. Stir for 0.5-1.0 hrs to ensure the EGDS has dissolved.
- v) Commence cooling cycle and ensure the emulsion is sufficiently agitated, c.a. 60-100 rpm (80 rpm is preferable).
- vi) When the product has cooled to 30-35°C, add remaining SLES and mix until homogeneous.
- vii) Check solids and adjust with water into specification.
- viii) Add preservative and adjust pH accordingly.

The concentrates require the following cooling cycle:

<u>Temperature</u>	<u>Cooling Rate</u>	<u>Time</u>
80 – 60°C	0.1°C min ⁻¹	3 hrs 20 mins
60 – 30°C	0.05°C min ⁻¹	10 hrs

Concentrates prepared using the above method were found to have a consistent pearl size, (20 to 40 microns) freeze/thaw stable and do not separate after one month at 40°C.

Example 1

<u>Component</u>	<u>Solids (%)</u>	<u>W/W (%)</u>
EMPICOL ESB 70 (70 %) Sodium laureth-2 sulphate	24	34.3
EMPILAN EGDS/A Ethylene glycol distearate	20	20
SODIUM CHLORIDE	3	3
PRESERVATIVE	q.s	q.s
WATER	-	Balance

Appearance: White/off white metallic pearl

pH (100 %): 5.5 – 6.5

Odour: Characteristic

Solids: 46 - 48 % (typical)

Viscosity @ 25°C: < 20000 cps

Density @ 20°C: 0.95 – 1.05g
cm⁻³**Example 2**

<u>Component</u>	<u>Solids (%)</u>	<u>W/W (%)</u>
EMPICOL ESB 3/MX (27 %) Sodium laureth-2 sulphate	14	51.9
EMPILAN EGDS/A Ethylene glycol distearate	26	26
SODIUM CHLORIDE	2	2
PRESERVATIVE	q.s	q.s
WATER	-	Balance

Appearance: White/off white metallic pearl

pH (100 %): 5.5 – 6.5

Odour: Characteristic

Solids: 40 - 42 % (typical)

Viscosity @ 25°C: < 20000 cps

Density @ 20°C: 0.95 – 1.05g
cm⁻³

Example 3

<u>Component</u>	<u>Solids (%)</u>	<u>W/W (%)</u>
EMPICOL ESB 70 (70 %) Sodium laureth-2 sulphate	20.5	29.3
EMPILAN EGDS/A Ethylene glycol distearate	26-27	26-27
SODIUM CHLORIDE	4.5	4.5
PRESERVATIVE	q.s	q.s
WATER	-	Balance

Appearance: White/off white metallic pearl

pH (100 %): 5.5 – 6.5

Odour: Characteristic

Solids: 51-54 % (typical)

Viscosity @ 25°C: < 10000 cps (typical)

Density @ 20°C: 0.95 – 1.05g
cm⁻³**Example 4**

<u>Component</u>	<u>Solids (%)</u>	<u>W/W (%)</u>
EMPICOL ESB 70 (70 %) Sodium laureth-2 sulphate	20.5	29.3
EMPILAN EGDS/A Ethylene glycol distearate	26 - 27	26 – 27
SODIUM CITRATE	10	10
PRESERVATIVE	q.s	q.s
WATER	-	Balance

Appearance: White/off white metallic pearl

pH (100 %): 5.5 – 6.5

Odour: Characteristic

Solids: 57 - 60 % (typical)

Viscosity @ 25°C: < 10000 cps (typical)

Density @ 20°C: 0.95 – 1.05g
cm⁻³

CLAIMS

1. The use of a protolamellar aqueous surfactant to suspend solid particles.
2. The use according to claim 1 of a protolamellar aqueous surfactant to suspend pearlisers.
3. A composition comprising protolamellar aqueous surfactant and suspended particles of pearliser.
4. A composition comprising water, from 17 to 30% by weight of alkali metal, ammonium or C_{1-6} amine salt of a C_{10-18} 1 to 10 mole ethoxy sulphate, sufficient electrolyte to form with said sulphate and water an optically isotropic phase which exhibits optically anisotropic properties and lamellar symmetry when subject to shear, from 15 to 60% by weight of particles of ethylene glycol mono and/or distearate having a particle size of from 6 to 60 microns suspended in said composition.
5. A composition comprising water, from 19 to 28% by weight of sodium C_{12-14} alkyl 1 to 5 mole ethoxy sulphate, from 18 to 28% by weight of pearliser consisting at least predominantly of ethylene glycol distearate, optionally in admixture with a minor proportion of ethylene glycol monostearate and having a particle size of from 10 to 50 microns and from 2 to 5% by weight of sodium chloride.
6. A method of making a pearliser suspension as claimed in any of claims 3 to 5 which comprises forming an emulsion of ethylene glycol distearate, optionally in admixture with a minor proportion of ethylene glycol monostearate, at a temperature above its melting point, in a solution of from 18 to 28% by weight of a sodium C_{10-18} alkyl 1 to 10 mole ethoxy sulphate and from 2 to 5% by weight of sodium chloride and cooling said emulsion.

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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- (71) Applicant (*for all designated States except US*): RHO-DIA CONSUMER SPECIALTIES LIMITED trading as ALBRIGHT & WILSON SURFACTANTS EUROPE [GB/GB]; 210-222 Hagley Road West, Oldbury, West Midlands B68 0NN (GB).
- (72) Inventors; and
- (75) Inventors/Applicants (*for US only*): HATCHMAN, Kevin [GB/GB]; 5 Byland Close, Friarscroft, Bromsgrove, Worcestershire B61 7PL (GB). CROMBIE, Richard, Llewelyn [GB/GB]; 201 Court Oak Road, Harborne, Birmingham B17 9AD (GB). HAWKINS, John [GB/GB]; 21 Chandler Avenue, Kinver, South Staffs DY7 6AG (GB).
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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 01/25378 A2

(54) Title: STRUCTURED SURFACTANT SYSTEMS

(57) Abstract: The protolamellar phase (a disc micellar, optically isotropic phase which exhibits transient anisotropy under stress) is capable of suspending solid particles. It is particularly effective for suspending pearlisers.

DECLARATION FOR PATENT APPLICATION

As an undersigned inventor, I hereby declare that:

My residence, post office address and country of citizenship are as stated directly below my name.

I believe (check one) ☐ I am the original, first and sole inventor
☒ I am a joint inventor and the below named inventors are the
original and first inventors

of the subject matter which is claimed and for which a patent is sought on the invention entitled
"STRUCTURED SURFACTANT SYSTEMS" (Attorney Docket **MPD309**),
the specification of which

(check one) ☐ is attached hereto.

☒ was filed on October 4, 2000

as Application Serial No. PCT/EP00/09677

and was amended on _____
(if applicable)

I further declare that I have reviewed and understand the contents of the above identified
specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the United States Patent and Trademark Office (hereinafter
"the Office") all information known to me to be material to patentability of the subject matter
which is claimed as defined in 37 C.F.R. §1.56.

I hereby claim foreign priority benefits under 35 U.S.C. §119 of any foreign application(s) for
patent or inventor's certificate indicated below and have also identified below any foreign
application for patent or inventor's certificate having a filing date before that of the application on
which priority is claimed:

Prior Foreign Application(s)		Day/Month/Year Filed	Priority Claimed	
Number	Country		Yes	No
<u>9923593.9</u>	<u>Great Britain</u>	<u>07 October 1999</u>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
_____	_____	_____	<input type="checkbox"/>	<input type="checkbox"/>
_____	_____	_____	<input type="checkbox"/>	<input type="checkbox"/>

I hereby claim the benefit under 35 U.S.C. §120 of any United States application(s) listed below
and, insofar as the subject matter of each of the claims of this application is not disclosed in the
prior United States application in the manner provided by the first paragraph of 35 U.S.C. §112,

33

(512) 483-0933 (Fax)

[illegible]

2-D

Date _____

Residence 201 Court Oak Road, Harborne, Birmingham B17 9AD Great Britain *GBN*

Citizenship British

Post Office Address Same

Full name of third inventor, if any *3-00* John Hawkins

Inventor's Signature

Glee Jen

14-3-2002

Date

Residence 21 Chandler Avenue, Kinver, South Staffordshire DY7 6AG Great Britain *GBN*

Citizenship British

Post Office Address Same

Full name of fourth joint inventor, if any

Inventor's Signature

Date

Residence

Citizenship

Post Office Address